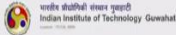


Biomass Conversion and Biorefinery
Prof. Kaustubha Mohanty
Department of Chemical Engineering
Indian Institute of Technology - Guwahati

Lecture – 30
Methanol Production and Utilization

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Module	Module name	Lecture	Content
10	Hydrogen, Methane and Methanol	03	Methanol production and utilization




Good morning students. Today is lecture 3 under module 10 and today we will be discussing about methanol production and utilization. So, let us begin.

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Introduction

- *Methanol* as an alternative biofuel for internal combustion and other engines has gained interest since last few years.
- Methanol is being used as fuel either in combination with gasoline or independently.
- Methanol, or methyl alcohol, is an organic compound, CH_3OH , with a molecular weight equal to 32.042 u.m.a.
- Now-a-days, it is one of the main competitors to *hydrogen* as clean fuel.
- The use of hydrogen appears to be the most promising from an energy point of view. In fact, it has the highest energy content per unit weight of any known fuel (142 kJ/g), and in comparison to the other known natural gases, it is environmentally safe.



Methanol as an alternative biofuel for internal combustion and other engines has gained interest since last few years. Methanol is being used as fuel either in combination with gasoline or


independently. Methanol or methyl alcohol is an organic compound CH_3OH with a molecular weight of 32.042. Nowadays, it is one of the main competitors to hydrogen as a clean fuel.

The use of hydrogen appears to be most promising from an energy point of view. In fact, it has the highest energy content per unit weight of any known fuel, 142 kilojoules per gram. It is a very big energy content you can say and in comparison to the other natural gases it is environmentally safe.

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- At present, the **problems** related to the use of hydrogen as a new energy resource are the *costs of purification processes and the difficulties linked to infrastructure for storage and transport.*
- The most important competitor appears to be *methanol*, which has an octane number of 113 and a density that is about half that of gasoline.
- Among several uses, *methanol can be mixed with conventional gasoline without requiring any technical modification in the vehicle fleet.* Most of the methanol-fueled vehicles use *M85, a mixture of 85% methanol and 15% unleaded gasoline.*
- Furthermore, methanol can be used as a convenient energy carrier for hydrogen storage and transportation, as an easily transportable fuel, and also in the chemical industry as a solvent and as a C1 building block for producing intermediates and synthetic hydrocarbons, including polymers and single-cell proteins.

Cifer and Bala, Energy Conversion and Management, 2017, 48 (2), 519-527



At present, the problems related to the use of hydrogen as a new energy resource are the costs of purification process and the difficulties linked to the infrastructure for storage and transport. You must be aware that huge work across the entire world is going on for hydrogen storage, still many things are unanswered and people are working day and night to make it successful. There are interesting works already being commercialized also.

One of the most important competitor appears to be methanol, which has an octane number of 113 and a density that is about half that of gasoline. Among several uses, methanol can be mixed with conventional gasoline without requiring any technical modification in the vehicle fleet. So, that is the most interesting part of methanol blending. So, most of the methanol fueled vehicles use M85.

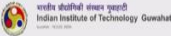
So M85 is nothing but a mixture of 85% methanol and 15% of unleaded gasoline. Furthermore, methanol can be used as a convenient energy carrier for hydrogen storage and transportation, as an easily transportable fuel, and also in the chemical industry as a solvent and as a C1

building block for producing intermediates and synthetic hydrocarbons including polymers and single-cell proteins. Methanol is an extremely valuable solvent for most of the chemical industries.

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- For all these reasons, methanol is considered to be the transition molecule from fossil fuels to renewable energies.
- It is only *slightly soluble in fat and oil* and represents one of the *most important chemical raw materials*.
- Indeed, the primary use of methanol is in the chemical industry, as either a feedstock, solvent, or cosolvent.
- Methanol is *less expensive* to produce in comparison with ethanol, however it has a *lower energy density and is more toxic*.
- Methanol may be made from *hydrocarbon or renewable resources*, in particular *natural gas and biomass* respectively. It can also be synthesized from *carbon dioxide and hydrogen*.
- For direct engine application, usually a *blend of methanol, ethanol and petroleum* is anytime preferred than individual fuels.

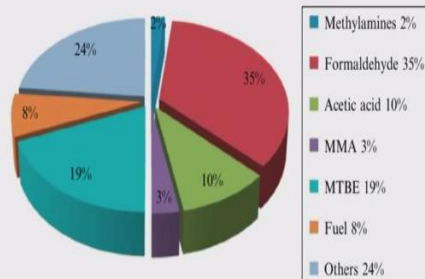
Bezzano and Maestri, Prog. Energy Combust. Sci. 2016, 58, 71-105.



For all these reasons, methanol is considered to be the transition molecule from fossil fuels to renewable energies. It is only slightly soluble in fat and oil and represents one of the most important chemical raw materials. Indeed, the primary use of methanol is in the chemical industry as either a feedstock solvent or a cosolvent. Methanol is less expensive to produce in comparison to ethanol (as a fuel when we talk about ethanol uses of fuel) however, it has a lower energy density and it is a little more toxic than ethanol. Now methanol may be made from hydrocarbons or renewable sources, in particular natural gas and biomass respectively. That is why we are discussing it under this course. It can also be synthesized from carbon dioxide and hydrogen. For direct engine application, usually a blend of methanol, ethanol and petroleum is anytime preferred than individual fuels.

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- Approximately 65% of the methanol produced worldwide is consumed for the production of acetic acid, methyl and vinyl acetates, methyl methacrylate, methylamines, methyl tert-butyl ether (MTBE), fuel additives, and other chemicals.
- The remaining portion is converted into formaldehyde and the resulting products, as shown in following figure.



Datta et al., In Methanol, 2010, 3-20

So, if you talk about the methanol produced and its use, approximately 65% of methanol produced worldwide is consumed for the production of acetic acid, vinyl and methyl acetates, methyl methacrylate, methylamines, MTBE that is methyl tert-butyl ether, fuel additives and other chemicals. The remaining portion is converted into formaldehyde and the resulting products as shown in the following figure. You can see that if you go by percentage formaldehyde is the highest.

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- Methanol was first produced by destructive distillation (*pyrolysis*) of wood and was known as *wood alcohol*.
- During the OPEC 1973 oil crisis, methanol was produced from coal as a proven fuel with well-established manufacturing technology and sufficient resources to replace gasoline.
- Later on, in 1986, Swedish Motor Fuel Technology Co. extensively reviewed the use of alcohols and alcohol blends as motor fuels. They reported in detail the methanol production from natural gas, very heavy oils, bituminous shales, coals, peat as well as biomass.
- Slowly then methanol's use as a fuel started gaining interest basically as a blend.
- In 2018, Govt. of India announced that if feasible, *passenger vehicles will run on 15% Methanol mixed fuel*. At present, vehicles in India use up to 10% ethanol-blended fuel.
- If approved by the government, it is estimated to cut monthly fuel costs at least by 10%.

So, methanol was first produced by destructive distillation or we can call it as pyrolysis of wood and was known as wood alcohol by the British scientists. During the OPEC 1973 oil crisis, methanol was produced from coal as a proven fuel with a well-established manufacturing technology and sufficient resources to replace gasoline. Later on in 1986 Swedish Motor Fuel

Technology Corporation extensively reviewed the use of alcohols and alcohol blends as motor fuels.

So, they reported in detail the methanol production from natural gas, very heavy oils, bituminous shales, coals, peat as well as biomass. Slowly then methanol's use as a fuel started gaining interest basically as a blend. In 2018, Government of India announced that if feasible, passenger vehicles will run on 15% methanol mixed fuel. At present vehicles in India use up to 10% of ethanol-blended fuel. If approved by the government it is estimated to cut monthly cost at least by 10%.

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Methanol From Natural Gas

- Many technologies have been developed over the years to produce methanol from different sources.
- In fact, it can be synthesized from several carbon-containing feedstocks, including natural gases (it could even be produced right at the gas well by oxidative transformation), coal, biomass, or CO₂, the latter directly recovered from the atmosphere.
- Currently, about 90% of methanol is produced from natural gas.
- The process route for the production of the simplest alcohol is relatively straightforward, involving the three following basic steps:
 - *Production of synthesis gas (syngas)*
 - *Conversion of the syngas into crude methanol*
 - *Distillation of the crude methanol to achieve the desired purity*

Illy et al., Springer, 2014, ISBN 978-1-441-99709-7

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Now we will discuss about the various ways to produce methanol. So, the first one is methanol from natural gas. Many technologies have been developed over the years to produce methanol from different sources. In fact, it can be synthesized from several carbon containing feedstocks including natural gases. It could even be produced right at the gas well by oxidative transformation - that means production at source, which looks good from the economical point of view, then coal, biomass or carbon dioxide, the latter directly recovered from the atmosphere.

Currently about 90% of methanol is produced from natural gas. The process route for the production of the simplest alcohol is relatively straightforward involving the three following basic steps. The first one is the production of syngas or synthesis gas, Second is the conversion of syngas into the crude methanol. Third and one of the most important aspect is of course distillation of the crude methanol to achieve the desired purity.

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- The process of converting syngas into crude methanol occurs at a pressure of 50-100 bar and a temperature of 200-300 °C. The main reactions involved in methanol synthesis are shown below.
- Hydrogenation of carbon monoxide: $\text{CO} + 2\text{H}_2 \rightleftharpoons \text{CH}_3\text{OH}$
- Divided into two consecutive steps: $\text{CO} + \text{H}_2 \rightleftharpoons \text{CH}_2\text{O}$ $\text{CH}_2\text{O} + \text{H}_2 \rightleftharpoons \text{CH}_3\text{OH}$
- Hydrogenation of carbon dioxide: $\text{CO}_2 + 3\text{H}_2 \rightleftharpoons \text{CH}_3\text{OH} + \text{H}_2\text{O}$
- In addition, the conversion of synthesis gas is subjected to a *thermodynamic equilibrium* that limits the process to a low conversion per pass and, therefore, implies a large recycle of unconverted gas.
- The resulting recycle and cooling duty are largely responsible for the investment costs of this process segment.
- Over the years, several *solid catalysts* have been developed in order to maximize methanol yield and selectivity and minimize byproduct formation.



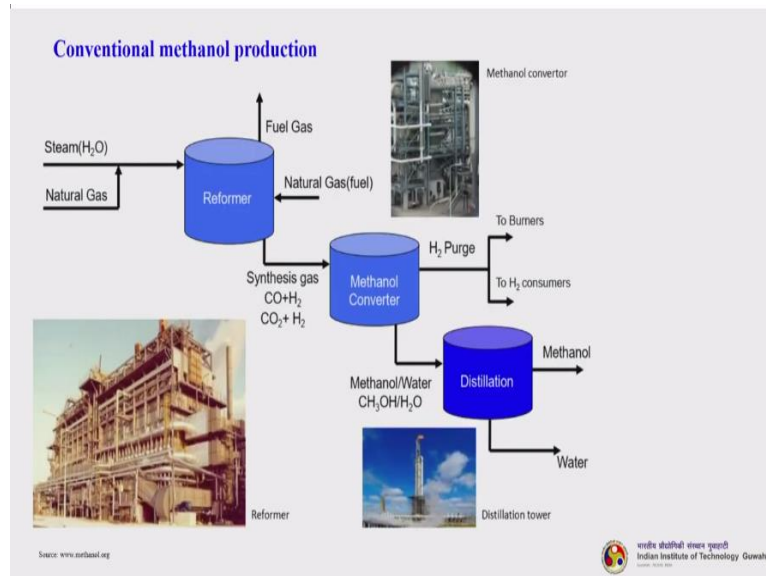
The process of converting syngas into crude methanol occurs at a pressure of 50 to 100 bar and a temperature of 200 to 300 degrees centigrade. The main reactions involved in methanol synthesis are shown below. The first one is hydrogenation of carbon monoxide. So carbon monoxide plus 2 hydrogen gives us methanol. Then it is divided into two consecutive steps. The carbon monoxide plus hydrogen gives us formaldehyde, formaldehyde plus hydrogen gives us methanol. Again the last one hydrogenation of carbon dioxide, so carbon dioxide plus 3 hydrogen gives us methanol plus water.

- Hydrogenation of carbon monoxide: $\text{CO} + 2\text{H}_2 \rightleftharpoons \text{CH}_3\text{OH}$
- Divided into two consecutive steps: $\text{CO} + \text{H}_2 \rightleftharpoons \text{CH}_2\text{O}$ $\text{CH}_2\text{O} + \text{H}_2 \rightleftharpoons \text{CH}_3\text{OH}$
- Hydrogenation of carbon dioxide: $\text{CO}_2 + 3\text{H}_2 \rightleftharpoons \text{CH}_3\text{OH} + \text{H}_2\text{O}$

So, you have to understand that these all reactions are happening simultaneously. So, in addition the conversion of synthesis gas is subjected to a thermodynamic equilibrium that limits the process to a lower conversion per pass and, therefore, implies a large recycle of the unconverted gas.

The resulting recycle and cooling duty are largely responsible for the investment cost of the process segment. Over the years, several solid catalyst have been developed in order to maximize methanol yield and selectivity and minimize byproduct formation.

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So, this is a conventional methanol production a schematic representation. So, we will try to understand in a nutshell the flow diagram, the block diagram basically. So, steam plus natural gas they are fed to a reformer. The first reformer looks something like this in industrial scale. So, you get a fuel gas here and you can also partly supply the natural gas or fuel. Then what you get is basically the syngas or synthesis gas, carbon monoxide plus hydrogen or carbon dioxide plus hydrogen.

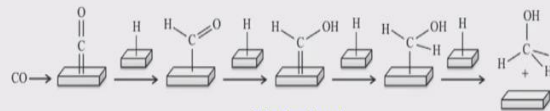
These are the two reactions that will happen inside the methanol converter. Now from the methanol converter, we will get the hydrogen purge. So, we can again purify this hydrogen and it can go to burners or to the hydrogen consumers depending upon what is the purity. And this is how it looks like a methanol converter in the real life industries.

Then the methanol water mixture it goes to a distillation unit (it looks something like this), where it is purified to get an almost 99% pure methanol and the leftover water can further be recycled to various processes.

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The BASF (Badische Anilin und Soda Fabrik- a German company) process : High-pressure method

- A great variety of oxides and metals have been tested as hydrogenation catalysts, excluding only the eighth group of the periodic system. All reactions were carried out at high pressure, 250-300 bar, and high temperatures, 320-450 °C. Among all, the two catalysts that gave the best results in these reaction conditions were $\text{ZnO/Cr}_2\text{O}_3$ and ZnO/CuO .
- On this basis, many studies have been done to investigate the reaction mechanisms of methanol production, in presence of the above-mentioned solid catalysts (heterogeneous catalysis).
- As shown below, two different ways were proposed, both involving the adsorption of CO and H_2 : the mechanism A shows that the reaction takes place in *four consecutive hydrogenation steps*.



King, Catal. Rev. Sci. Eng., 1980, 22(2), 235-270
Debraj et al., In Methanol, 2018, 3-28

Mechanism A



And next is the BASF. It is a German company developed process. It is a high pressure method. Now, a great variety of oxides and metals have been tested as hydrogenation catalysts excluding only the eighth group of the periodic system, almost all have been used. So, all reactions were carried out at high pressure 250 to 300 bar and high temperature 320 to 450 degrees centigrade.

So among all, two catalysts which has given a very extremely high selectivity and the best results in these reaction conditions were zinc oxide/ Cr_2O_3 and zinc oxide/copper oxide catalysts. So bimetallic catalyst basically. So, on this basis many studies have been done to investigate the reaction mechanisms of methanol production in presence of the above mentioned solid catalysts.

These are solid catalysts because these heterogeneous catalysts are easy to recover from the liquid phase stream and you can regenerate and reuse it. Homogeneous catalysts we cannot do it. So, as shown below two different ways are proposed both involving the adsorption of carbon monoxide and hydrogen on the surface of the catalyst. So, this is the mechanism A and the second one is shown in the next slide.

So, the mechanism A shows that the reaction takes place in four consecutive hydrogenation steps. So, there are four consecutive hydrogenation steps. You understand that this is the surface of the catalyst, carbon monoxide, then hydrogen is getting attached on the surface or adsorbed on the surface of the catalyst. So, in four subsequent steps so you get methanol.

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- In mechanism B *both CO and a hydroxyl group on the catalyst surface* are involved.
- The first step takes place by an insertion of carbon monoxide to form a formate intermediate; subsequent hydrogenation and dehydration lead to the formation of methanol, passing through a methoxide intermediate.

Mechanism B

- It is noteworthy that the *two mechanisms proposed differ not only for the intermediates formed, but also for the way in which they are bonded to the catalyst surface*, that is with a carbon atom in mechanism A and with oxygen in B. As a result, catalytic cycles of ZnO/Cr₂O₃ and ZnO/CuO catalysts were proposed.

Datta et al., In Methanol, 2016, 5-28

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Then in the second one, this is mechanism B. Here in the mechanism B, both carbon monoxide and a hydroxyl group on the catalyst surface are involved. So, in the mechanism A only carbon monoxide and hydrogen were involved, here one carboxylic group, you can see that C double bond O, this carboxylic group is coming into picture and it is getting adsorbed on the surface of the catalyst and then the reaction proceeds in this way.

So, the first step, it takes place by insertion of a carbon monoxide to (form a) formate intermediate. So, the formate intermediate is this COOH. Subsequent hydrogenation and dehydration leads to the formation of methanol passing through a methoxide intermediate. Now, it is noteworthy that these two mechanisms proposed, differ not only for the intermediates formed, but also for the way in which they are bonded to the catalyst surface, that is with a carbon atom in the mechanism A and with oxygen in mechanism B. So, that is the basic difference between these two mechanisms. As a result, catalytic cycles of the zinc oxide /Cr₂O₃ and zinc oxide/copper oxide catalyst were proposed and they have been found to be extremely selective.

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The ICI (Imperial Chemical Industries) process : Low-pressure method

- In the 1960s, the BASF high-pressure method was overcome by a low-pressure method created by ICI (now Johnson Matthey). They developed a new route for methanol synthesis in a pressure range equal to 35-54 bar and at temperatures ranging from 200 °C to 300 °C.
- This was made possible not only by the discovery of a new, more active and selective *copper-based catalyst (Cu/ZnO/Al₂O₃)*, but also by the *development of new advanced purification processes for synthesis gases*, which allowed them to use sulfur-chlorine-free syngas.
- Despite the fact that the catalytic power of copper/zinc catalysts in methanol synthesis was already well known, this was not exploited commercially due to their low lifetime and low thermal stability, mostly caused by their *deactivation by poisoning*.
- These problems were overcome thanks to the addition of alumina, which increases the stability of the Cu/ZnO catalyst and inhibits the thermal formation of Cu crystallites.

Urban et al. | Molecules 2016, 7, 91



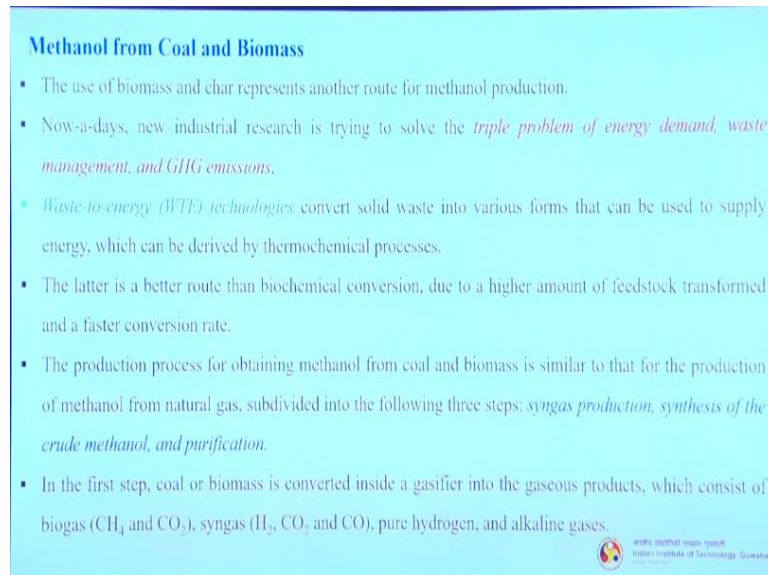
So, the next is the Imperial Chemical Industries process which is a low-pressure method. In the 1960s, the BASF high pressure method was overcome by a low-pressure method created by ICI. Now it is Johnson Matthey. So, they developed a new route for methanol synthesis in a pressure range equal to 35 to 54 bar, much lower than that of the BASF method, and a temperature ranging from 200 to 300 degrees centigrade, again a lower temperature range than that of the BASF.

Now, this was made possible not only by the discovery of a new more active and selective copper based catalyst (copper/zinc oxide/alumina Al₂O₃), but also by the development of a new advanced purification process for synthesis gases, which allowed them to use sulfur-chlorine-free syngas. Now, I have told you many times that any such processes when you talk about the biomass conversion into value added products; purification step after the product is formed, either that is in the gaseous phase or in the liquid phase maybe sometimes in solid phase also, the purification phase is the most time consuming, energy consuming as well as it is a costly affair. Almost 40% of the product cost goes to the purification. So, if you have an excellent purification process which is economical, then the product price will come down and as well as you will get a higher purity product.

That is what it has happened in the ICI method. Despite the fact that the catalytic power of copper zinc catalyst in methanol synthesis was already well known. This was not exploited commercially due to their low lifetime and the low thermal stability mostly caused by their deactivation by poisoning. So, very quickly they get deactivated, so that is once they deactivate we call them catalyst poisoning, so they are no more taking part in the reaction.

Now, these problems were overcome thanks to the addition of alumina, which increases the stability of the copper zinc oxide catalysts and inhibits the thermal formation of the copper crystallites.

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Methanol from Coal and Biomass

- The use of biomass and char represents another route for methanol production.
- Now-a-days, new industrial research is trying to solve the *triple problem of energy demand, waste management, and GHG emissions*.
- *Waste-to-energy (WTE) technologies* convert solid waste into various forms that can be used to supply energy, which can be derived by thermochemical processes.
- The latter is a better route than biochemical conversion, due to a higher amount of feedstock transformed and a faster conversion rate.
- The production process for obtaining methanol from coal and biomass is similar to that for the production of methanol from natural gas, subdivided into the following three steps: *syngas production, synthesis of the crude methanol, and purification*.
- In the first step, coal or biomass is converted inside a gasifier into the gaseous products, which consist of biogas (CH_4 and CO_2), syngas (H_2 , CO_2 and CO), pure hydrogen, and alkaline gases.

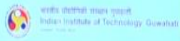
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So the next and most important process is of course methanol from coal and biomass. The use of biomass and char represent another route for methanol production. Nowadays, new industrial research is trying to solve the triple problem of energy demand, waste management and greenhouse gas emissions. Waste to energy technologies convert solid waste into various forms that can be used to supply energy which can be derived by the thermochemical processes.

The latter is a better route than biochemical conversion due to a higher amount of feedstock transfer and a faster conversion rate. The production process for obtaining methanol from coal and biomass is similar to that for the production of methanol from natural gas, subdivided into the following 3 steps. Syngas production, synthesis of the crude methanol and purification.

In the first step, coal or biomass is converted inside a gasifier into the gaseous products which consists of biogas that is methane and carbon dioxide, syngas (hydrogen, carbon dioxide and carbon monoxide), pure hydrogen and maybe some alkaline gases.

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- The gasification process is a thermochemical conversion technique that allows the conversion of solid biomass into gaseous mixtures with the help of gasifying agents such as air/oxygen, steam, and flue gases.
 - This process, using high temperatures in the presence of O₂, aims for a large-scale development to overcome the current limits (i.e., *low hydrogen production and high tar content in the syngas*).
 - In fact, the unwanted tar may cause the formation of tar aerosols and polymerization into more complicated structures, which are not favorable for hydrogen production through steam reforming.
 - For this reason, several research groups have focused their studies on *maximizing the hydrogen quantity produced and minimizing that of tar*.
 - Under typical gasification conditions, *oxygen levels are restricted to less than 30% of that required for complete combustion*, and CO and H₂ are the major products.
 - Methanol is produced from syngas utilizing conventional gasification of biomass at high temperatures (800-1000 °C); it can be made with any resource containing carbon, such as coal, or solid wastes (SW).
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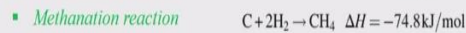
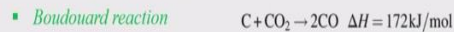
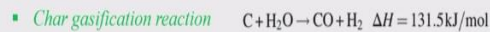
Now, the gasification process is a thermochemical conversion technique which we have discussed in detail in our previous classes that allows the conversion of solid biomasses into gaseous mixtures with the help of gasifying agent. It can be air or oxygen, steam and flue gases. Now, this process using high temperatures in the presence of oxygen aims for a large scale development to overcome the current limits that is the low hydrogen production and high tar content in the syngas.

In fact, the unwanted tar may cause the formation of tar aerosols and polymerization into more complicated structures, which are not favorable for hydrogen production through steam reforming. For this reason, several research groups have focused their studies on maximizing the hydrogen quantity produced and minimizing that of tar.

Under typical gasification conditions, oxygen levels are restricted to less than 30% of that required for complete combustion and carbon monoxide and hydrogen are the major products. Methanol is produced from syngas utilizing conventional gasification of biomass at high temperatures almost at 800 to 1000 degrees centigrade and it can be made with any resource containing carbon such as coal or solid wastes.

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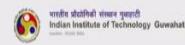
The conversion of gaseous products involves many reactions like



▪ However, conventional gasification processes applied to biomass **do not always produce a syngas with the quality required for methanol synthesis**. One of the **biggest problems** in the gasification of biomass is the **formation of tar and char** via reduction of carbon oxides.

▪ The synthesis gas for methanol production should only contain a small proportion of inert gas components.

▪ In fact, the use of air as a gasification agent results in a syngas with a high nitrogen content.



Now, we will look for the reactions. The conversion of gaseous products involve many reactions. So 5 main reactions have been listed here. The char gasification reaction, so carbon plus water gives carbon monoxide plus hydrogen. Then water shift gas reaction, so carbon monoxide plus water gives carbon dioxide plus hydrogen. Steam reforming of methane, methane plus water gives carbon monoxide plus 3 hydrogen. Boudouard reactions, so carbon plus carbon dioxide reacts to give us 2 moles of carbon monoxide. Then methanation reaction, here the carbon plus 2 hydrogen gives us methane.

- Char gasification reaction $C + H_2O \rightarrow CO + H_2 \quad \Delta H = 131.5 \text{ kJ/mol}$
- WGS reaction $CO + H_2O \rightleftharpoons CO_2 + H_2 \quad \Delta H = -41 \text{ kJ/mol}$
- Steam reforming of methane $CH_4 + H_2O \rightleftharpoons CO + 3H_2 \quad \Delta H = 206 \text{ kJ/mol}$
- Boudouard reaction $C + CO_2 \rightarrow 2CO \quad \Delta H = 172 \text{ kJ/mol}$
- Methanation reaction $C + 2H_2 \rightarrow CH_4 \quad \Delta H = -74.8 \text{ kJ/mol}$

Now again the same methane gets converted to carbon monoxide. So, all these are going on simultaneously. However, unconventional gasification processes applied to biomass do not always produce a syngas with the quality that is required for methanol synthesis.

So, please note that any syngas that is produced through biomass gasification cannot be used to convert it to methanol, we need certain purity. So, one of the biggest problem in the gasification of biomass is the formation of tar and char via the reduction of the carbon oxides. Now, the synthesis gas for methanol production should only contain a small proportion of inert gas components.

In fact, the use of air as a gasification agent always results in a syngas with a high nitrogen content which is not actually desirable when you talk about methanol conversion.

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- For methanol synthesis the optimal $H_2:CO_2$ molar ratio in the syngas is >2 and then the gasification of biomass always results in a gas with a too low $H_2:CO_2$ ratio.
- Usually the *WGS (Water gas shift)* process is the most frequently used process for ensuring a suitable ratio of $CO_2:CO:H_2$ and mainly to convert the CO into CO_2 .
- If CO is present when the syngas is combusted in the gasification, the CO_2 removal efficiency will be limited since the CO in syngas will be converted to CO_2 during the combustion process.
- The reaction temperature for the WGS is $375\text{ }^\circ\text{C}$ and the pressure is 40 bar. *Carbon monoxide is converted to carbon dioxide and hydrogen.*
- The carbon dioxide produced during the WGS process must be separated from the syngas in order to ensure a suitable ratio of $CO_2:CO:H_2$ for the commercially available methanol production catalyst required to be 5:28:63.
- Sulfur must be removed from the syngas as sulfur is very hazardous for the catalyst used for methanol production and WGS reaction.

Spectra and Bhask. Centre for Solar Energy and Hydrogen Research (CSHR), 1999.



So, for methanol synthesis the optimal hydrogen carbon dioxide molar ratio in the syngas is greater than 2. And then the gasification of biomass always results in a gas with a too low hydrogen to carbon dioxide ratio. So, this is a problem with biomass to methanol. So, usually the water gas shift process is the most frequently used process for ensuring a suitable ratio of carbon dioxide carbon monoxide hydrogen and mainly to convert the carbon monoxide to carbon dioxide.

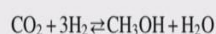
If carbon monoxide is present when the syngas is combusted in gasification, the carbon dioxide removal efficiency will be limited since the carbon monoxide in syngas will be converted to carbon dioxide during the combustion process. The reaction temperature for the water gas is 375 degrees centigrade and the pressure is 40 bar. Carbon monoxide is converted to carbon dioxide and hydrogen.

The carbon dioxide produced during the water gas shift process must be separated from the syngas in order to ensure a suitable ratio of the carbon dioxide carbon monoxide hydrogen for the commercially available methanol production catalyst required minimum to be 5:28:63 in that ratio. So, sulfur must be removed from the syngas as sulfur is very hazardous for the catalyst used for methanol production as well as during the water gas shift reaction.

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Methanol from Catalytic Hydrogenation of CO₂

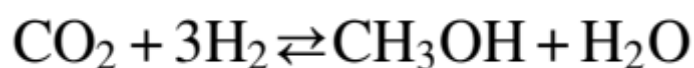
- CO₂ is a linear molecule that is *very stable* and *needs extra efforts to make it reactive*.
- Owing to its *high stability* ($\Delta G^\circ = -400 \text{ kJ mol}^{-1}$), a substantial energy input, optimized reaction conditions, and a catalyst with high stability and activity are required for converting CO₂ into value-added chemicals.
- Converting one mole of CO₂ to methanol requires an energy input of about 228 kJ and six electrons to reduce C⁴⁺ of CO₂ to C²⁻ of methanol.
- The carbon-oxygen bonds are very strong, and high energy is required for breaking them. For this reason, in order to convert CO₂ into methanol, *a good catalytic system is required*.
- The reaction of this catalytic conversion of CO₂ into methanol by hydrogenation is expressed by:



So, now we will discuss about the methanol from catalytic hydrogenation of carbon dioxide. Carbon dioxide is a linear molecule that is very stable and needs extra efforts to make it reactive. Owing to its high stability, a substantial energy input, optimized reaction conditions and a catalyst with high stability and activity are required for converting carbon dioxide into value-added chemicals.

Converting 1 mole of carbon dioxide to methanol requests an energy input of about 228 kilojoules and 6 electrons to reduce the carbon 4+ of carbon dioxide to C 2- of methanol. So, the carbon oxygen bonds are very strong and high energy is required for breaking them. For this reason in order to convert carbon dioxide into methanol, a very good catalytic system is the utmost need.

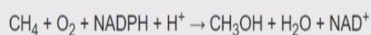
So, the reaction of this catalytic conversion of carbon dioxide into methanol by hydrogenation is given below. So, carbon dioxide plus 3 moles of hydrogen will give us methanol plus water.



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Methanol via Biosynthesis

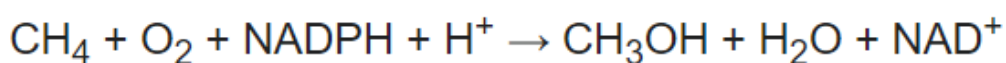
- The catalytic conversion of methane to methanol is effected by enzymes including *methane monooxygenases*. These enzymes are mixed-function oxygenases, i.e. oxygenation is coupled with production of water and NAD⁺.



- Both Fe- and Cu-dependent enzymes have been characterized. Intense but largely fruitless efforts have been undertaken to emulate this reactivity.



The next one is methanol biosynthesis way. The catalytic conversion of methane to methanol is effected by enzymes including methane monooxygenase. So these enzymes are mixed-function oxygenases, that is, oxygen is coupled with the production of water and NAD⁺. So this is the reaction: methane + oxygen + NADPH + hydrogen gives us methanol + water + NAD⁺.



Both iron and copper dependent enzymes have been characterized. Intense but largely fruitless efforts have been undertaken to emulate this reactivity because of various process optimization things as well as the yield.

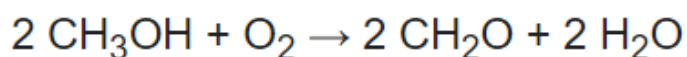
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Methanol to formaldehyde, acetic acid and methyl tert-butyl ether (MTBE)

- Methanol is primarily converted to formaldehyde, which is widely used in many areas, especially polymers.
- The conversion entails oxidation: $2 \text{CH}_3\text{OH} + \text{O}_2 \rightarrow 2 \text{CH}_2\text{O} + 2 \text{H}_2\text{O}$
- Methanol and isobutene are combined to give methyl tert-butyl ether (MTBE). *MTBE is a major octane booster in gasoline.*
- Acetic acid can be produced from methanol via following *Cativa™ process*.
- The Cativa process is a method for the production of acetic acid by the *carbonylation of methanol*.
- This process is similar to the Monsanto process and was developed by BP Chemicals.
- It is based on an *Iridium-containing catalyst*, such as the complex $[\text{Ir}(\text{CO})_2\text{I}_2]^-$



Now we will talk about the various uses of methanol. So the first one and one of the most important is of course methanol to various platform chemicals such as formaldehyde, acetate or acetic acid and MTBE - methyl tert-butyl ether. So methanol is primarily converted to formaldehyde which is widely used in many areas, especially polymers. The conversion entails the oxidation reaction 2 moles of methanol plus oxygen will give us 2 moles of formaldehyde and water.



So, methanol and isobutene are combined to give MTBE the methyl tert-butyl ether. So, MTBE is a very important class of chemical and it is a major octane booster that is getting used in gasoline. So acetic acid can be produced from methanol via following the Cativa process. It is a very well known process and has been well adapted also. The Cativa process is a method for the production of acetic acid by the carbonylation of methanol.

The process is similar to the Monsanto process and was developed by the BP chemicals. It is based on an Iridium containing catalyst such as this complex $\text{Ir}(\text{CO})_2\text{I}_2$ complex, so it is the Iridium based catalyst.

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- ❖ Methanol is converted to *methyl iodide*.
- ❖ Reaction of *methyl iodide* with the square planar active catalyst species (1) to form the octahedral iridium(III) species (2).
- ❖ This *oxidative addition reaction* involves the formal insertion of the iridium (I) centre into the carbon-iodine bond of methyl iodide.
- ❖ After ligand exchange (*iodide for carbon monoxide*), the migratory insertion of carbon monoxide into the iridium-carbon bond, step (3) to (4), results in the formation of a *square pyramidal species with a bound acetyl ligand*.

Joon, J.H. *Parsons Meth Rev*; 2000, 44(3), 94-101

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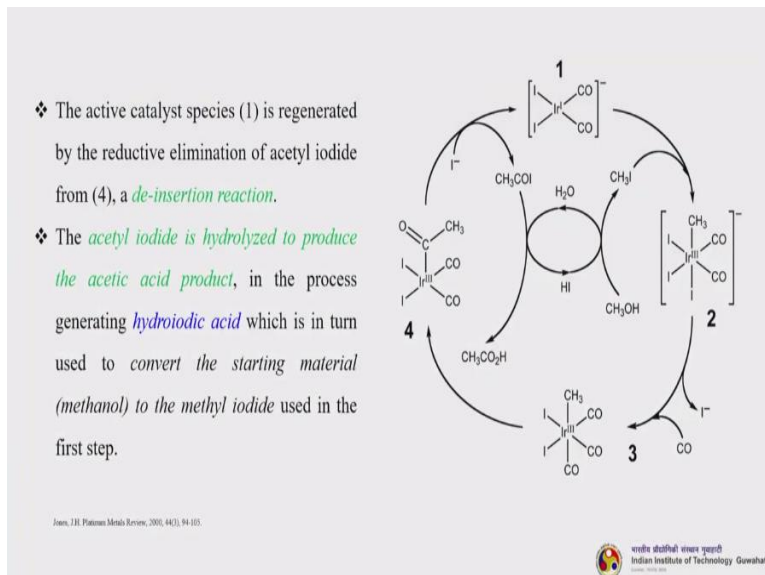
So let us see how it happens. I will explain you in the next two slides. You can just have a look at this particular cycle how it is happening, I will explain this. So, please look at the first second third and fourth four different intermediate steps. So, the base molecule is nothing but methanol from where we are producing various products. So this is your starting material methanol. We will come back to that inner cycle later on. So, let us start with the one.

So methanol is converted to methyl iodide. Methanol is getting converted to methyl iodide how it is I will explain it in the next slide. So, methyl iodide is the starting reaction you can say that. So, the reaction of methyl iodide with the square planar active catalyst species 1. So this is the species 1 with iridium in the center to form octahedral iridium 3 species in the section 2 you can say here. Again you can see that Ir 3.

Now, the next is this oxidative addition reaction involves the formal insertion of the iridium center into the carbon-iodine bond of the methyl iodide. So, after the ligand exchange, that means iodine for carbon monoxide, so in the next phase what is happening here iodide is getting replaced with a carbon monoxide. So, you can see that one more carbon monoxide is coming here.

The migratory insertion of carbon monoxide into the iridium based carbon bond in the step 3 to step 4 results in the formation of a square pyramidal species with a bound acetyl ligand. So, this is a square pyramidal species with acetyl ligand here C double bond O.

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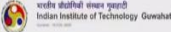
Now, the active catalyst species 1 is regenerated by the reductive elimination of acetyl iodide from 4 and via a de-insertion reaction. So, you can see that I- is getting removed from here and this particular complex is getting converted to the first species, which is the starting reaction species. Now, the acetyl iodide is hydrolyzed to produce the acetic acid product in the process generating hydroiodic acid, which is in turn used to convert the starting material that is methanol to the methyl iodide used in the first step. You can see how it is happening and

formerly you are getting your product of interest. This is the inner circle where this particular pathway is responsible for forming methyl iodide from methanol. So, this is the process detail.

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Methanol to Dimethyl ether (DME)

- In the last 10 years, one of the most promising technologies is the use of methanol as a C1 building block in the petrochemical industry, and a wide part of its production is consumed in the manufacturing of *DME as an alternative fuel*.
- DME has an *octane number and ignition temperature close to that of diesel fuel*.
- It leads to lower NO_x emissions, less smoke, and less engine noise than conventional diesel engines and, furthermore, can be easily transported.
- DME can also be used as a *chemical feedstock* for manufacturing many products, such as short olefins (ethylene and propylene), gasoline, hydrogen, acetic acid, and dimethyl sulfate.

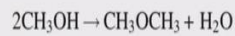


So, the next is methanol to DME - dimethyl ether. In the last 10 years, one of the most promising technologies is the use of methanol as a C1 building block in the petrochemical industry and a wide part of its production is consumed in the manufacturing of DME as an alternate fuel. Now, DME has the an octane number and ignition temperature close to that of diesel fuel that is why it is so important.

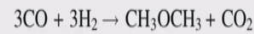
Now, it leads to lower NO_x emission, less smoke, less engine noise than the conventional diesel engine and furthermore can easily be transported. DME can also be used as a chemical feedstock for manufacturing of many products such as short olefins, for example ethylene and propylene, gasoline, hydrogen, acetic acid and dimethyl sulfate.

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- The current DME manufacturing process is a *double-step (indirect) synthesis*, in which methanol synthesis, carried out by one of the above-mentioned technologies, is followed by dehydration.



- Another developing process involves the *direct synthesis of DME from syngas*. In this process, syngas CO and H₂, with a 1:1 molar ratio, are converted in DME in the presence of a catalyst.



So, the current DME manufacturing process is a double-step indirect synthesis one in which methanol synthesis carried out by one of the above mentioned technologies is followed by dehydration, 2 moles of methanol gives us CH₃OCH₃ plus water.



Another developing process involves the direct synthesis of DME from syngas. Now, in this process syngas carbon monoxide and hydrogen with 1 : 1 molar ratio are converted to DME in the presence of a catalyst. So 3 moles of carbon monoxide plus 3 moles of hydrogen in the same ratio will give us DME plus carbon dioxide.

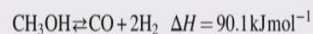


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Hydrogen Production

Methanol Decomposition

- Methanol decomposition (MD) is an *endothermic reaction* to produce H₂ and CO.

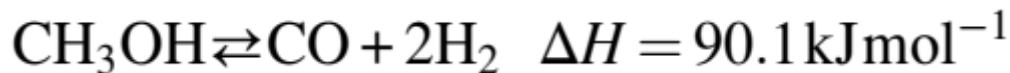


- This is applicable to the recovery of waste heat of around 200 °C from industries.
- For the development of heat-recovery systems, new catalysts that can be active even at temperatures below 200 °C are indispensable.
- *Usami et al. (1998)*, for example, have tested 15% wt Pd/ZrO₂, Pd/Pr₂O₃, and Pd/CeO₂ catalysts. These catalysts, prepared by co-precipitation, can produce high catalytic activity in the selective decomposition of methanol to CO and H₂ at a temperature as low as 200 °C.

Usami et al. Appl. Catal. A, 1998, 171 (1), 123-130.



Now hydrogen production from methanol, we will discuss a few processes. The first one is methanol decomposition. So, methanol decomposition is an endothermic reaction to produce hydrogen and carbon monoxide from methanol. So, methanol is giving us $\text{CO} + 2\text{H}_2$ you can see that ΔH is 90.1 kilojoules per mole.



And this is applicable to the recovery of waste heat of around 200 degrees centigrade from industries. For the development of a heat-recovery system, new catalysts that can be active even at temperatures below 200 degrees centigrade are indispensable. Usami et al in 1998 for example have tested 15% wet palladium/zirconium oxide, then Pd/Pr₂O₃ and PdCeO₂ catalyst.

Now, these catalysts, prepared by coprecipitation can produce high catalytic activity in the selective decomposition of methanol to carbon monoxide and hydrogen at a temperature as low as 200 degrees centigrade. It is a beautiful work, the reference is given here, you can read more if you are interested.

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Methanol-Water Solution Electrolysis

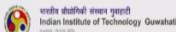
- Another way to produce hydrogen from methanol is by *methanol-water solution electrolysis* using an electrolytic cell.
- Generally, electrolysis of water is the best option for producing very pure hydrogen very quickly.
- As in the electrolysis of water, in the methanol-water system the *hydrogen produced is very pure* (hydrogen concentration is 95.5–97.2 mol%), but the theoretical voltage of the system is much lower than in water electrolysis (1.23 V in water electrolysis compared to 0.03 V in methanol-water solution electrolysis).
- In methanol-water solution electrolysis, hydrogen is produced by applying DC voltage to the electrolytic cell.

The next one is methanol-water solution electrolysis. Another way to produce hydrogen from methanol is by methanol-water solution electrolysis using an electrolytic cell. Now, generally electrolysis of water is the best option for producing very pure hydrogen and that too very quickly. As in the electrolysis of water, in the methanol-water system the hydrogen produced is very pure almost 95.5 to 97.2 mol percent.

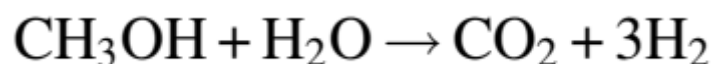
But the theoretical voltage of the system is much lower than that of the water electrolysis process, 1.23 volts in water electrolysis compared to 0.03 volt (very less it is in methanol-water solution electrolysis). So, in methanol-water solution electrolysis, hydrogen is produced by applying DC voltage to the electrolytic cell.

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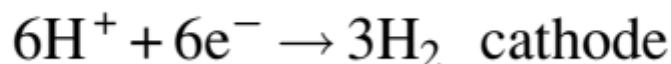
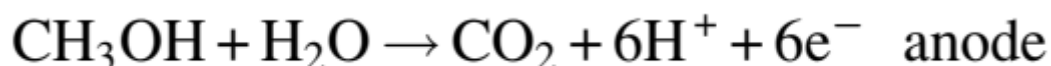
- In this system *the overall reaction* is: $\text{CH}_3\text{OH} + \text{H}_2\text{O} \rightarrow \text{CO}_2 + 3\text{H}_2$
- The reaction at the anode and at the cathode are:
$$\text{CH}_3\text{OH} + \text{H}_2\text{O} \rightarrow \text{CO}_2 + 6\text{H}^+ + 6\text{e}^- \quad \text{anode}$$
$$6\text{H}^+ + 6\text{e}^- \rightarrow 3\text{H}_2 \quad \text{cathode}$$
- At the **anode**, methanol reacts with water to produce carbon dioxide (exhausted outside the anode), protons, and electrons.
- The protons produced by the anode reaction move to the cathode of the electrolytic cell through the *proton exchange membrane (PEM)* and the electrons produced by the anode reaction move to the cathode through the external circuit containing the DC power supply.



So, in this system the overall reaction can be written as that methanol plus water gives us carbon dioxide plus 3 moles of hydrogen.



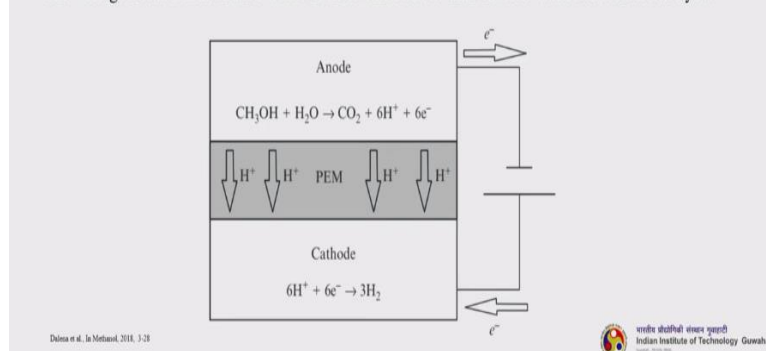
The reaction at anode and cathode are like this: methanol plus water is carbon dioxide + 6 proton + 6 electron that is in anode, then 6 proton + 6 electrons gives us 3 moles of hydrogen at cathode.



Now, at the anode methanol reacts with water to produce carbon dioxide, so exhausted outside the anode basically and protons and electrons. Now, the protons produced by the anode reaction move to the cathode of the electrolytic cell through the proton exchange membrane. There is a PEM membrane in between and the electrons produced by the anode reaction move to the cathode through the external circuit containing the DC power supply. This is how it happens.

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- At the **cathode**, protons supplied from the anode react with electrons supplied from the anode.
- Hydrogen production by methanol-water solution electrolysis is suitable for *portable power applications* because methanol-water solution electrolysis can start up and shut down in a moment and can produce hydrogen at a low temperature.
- The voltage needed in methanol-water is three times lower than that for water solution electrolysis.



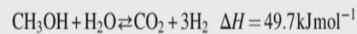
So you can imagine, it is a schematic representation simplified one. You can see that this is anode part, this is a cathode part and here this is your PEM - proton exchange membrane, it is a membrane, solid membrane. Now, you can see that when in the anode these reactions are happening, the protons are passing through the proton exchange membrane PEM membrane to the cathode side where it is combined with the electrons and resulting in hydrogen.

So, at the cathode protons supplied from the anode react with electrons supplied from the anode. Hydrogen production by methanol-water solution electrolysis is suitable for only portable power applications because methanol-water solution electrolysis can start up and shut down in a moment and can produce hydrogen at a lower temperature, very fast quick, but for the smaller applications. The voltage needed in methanol-water is three times lower than that of the water solution electrolysis.

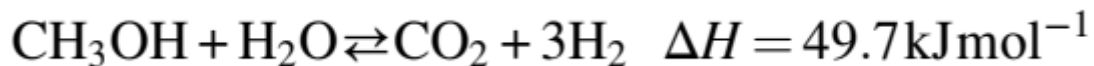
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Methanol Steam Reforming

- The production process that is much used in the production of hydrogen is the *methanol steam reforming (MSR)*.
- Compared to other fuels, methanol presents several advantages for hydrogen production.
- In fact, the absence of a strong C-C bond facilitates the reforming at low temperatures (200–300 °C), a range of temperatures that is very low when compared to other common fuels (800–1000 °C for methane and 400 °C for ethanol).
- The overall reaction of the MSR to the production of H₂ is expressed in



So, next one is methanol steam reforming. The production process that is much used in the production of hydrogen is the methanol steam reforming, popularly known as the MSR pathway. Now compared to the other fuels, methanol presents several advantages for hydrogen production. In fact, the absence of a strong carbon-carbon bond facilitates the reforming at low temperatures, a range of temperature that is very low when compared to other common fuels. The overall reaction for the MSR to the production of hydrogen is expressed as methanol plus water is carbon dioxide plus 3 moles of hydrogen. The delta H is a little lower here compared to other reaction, 49.7 kilojoules per mol.



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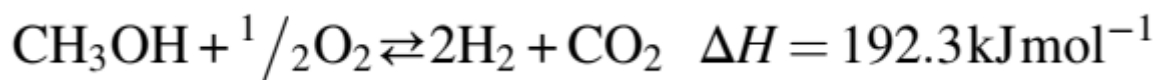
Partial Oxidation/Autothermal Reforming

- Unlike MD and MSR, *partial oxidation of methanol (POM)* is an exothermic reaction, so no additional heat is needed. However, the temperature control can be difficult.
- The addition of steam into POM leads to the ATR of methanol. The addition of steam in ATR intensifies H₂ production but lowers the reaction temperature and thereby the reaction rate.
- In contrast to MSR and methanol ATR, relatively less H₂ is produced from POM.
- POM is expressed as: $\text{CH}_3\text{OH} + \frac{1}{2}\text{O}_2 \rightleftharpoons 2\text{H}_2 + \text{CO}_2 \quad \Delta H = 192.3 \text{kJmol}^{-1}$
- By virtue of more heat released from POM being employed for chemical reactions, *POM is kinetically faster than MSR and methanol ATR*, implying that a smaller reactor is achievable for POM.



Then the next is partial oxidation or ATR autothermal reforming. So unlike MD and MSR, partial oxidation of methanol is an exothermic reaction. So, no additional heat is needed. However, the temperature control can become difficult. The addition of steam into POM leads to the ATR of methanol. Now, the addition of steam in ATR intensifies hydrogen production, but it lowers the reaction temperature and thereby the reaction rate. In contrast to MSR and methanol ATR, relatively less hydrogen is produced from POM.

The POM can be expressed as methanol plus half oxygen is 2 moles of hydrogen plus carbon dioxide with a delta H 192.3 kilojoules per mol and it is a higher delta H you can see if you compared to other reactions.




Now by virtue of more heat released from POM being employed for chemical reactions, POM is kinetically faster than the MSR and methanol ATR implying that a smaller reactor is achievable or it will be good for carrying out the POM.

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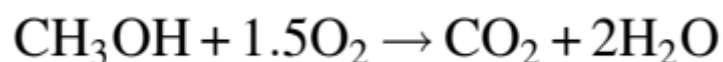
Methanol Fuel Cells

- One type of proton exchange membrane fuel cell (PEMFC) is the *Direct Methanol Fuel Cell (DMFC)* that uses methanol or methanol solutions as fuel and works at an ambient temperature.
- The overall reaction is: $\text{CH}_3\text{OH} + 1.5\text{O}_2 \rightarrow \text{CO}_2 + 2\text{H}_2\text{O}$
- The structure of the DMFC consists of two porous electrocatalytic electrodes on both sides of a solid polymer electrolyte membrane. The thermodynamic reversible potential for the overall cell reaction is 1.214 V.
- *Methanol and water are oxidized in the anode catalyst layer (ACL) and release electrons and protons, according to reaction.*

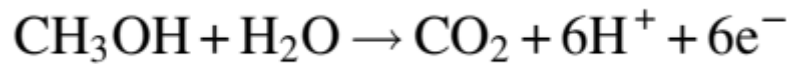
$$\text{CH}_3\text{OH} + \text{H}_2\text{O} \rightarrow \text{CO}_2 + 6\text{H}^+ + 6\text{e}^-$$


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Now, we will discuss about the methanol fuel cells. So, one type of proton exchange membrane fuel cell is the DMFC which is direct methanol fuel cell; that uses methanol or methanol solutions as fuel and works at an ambient temperature. Now, this is the reaction. So methanol plus 1.5 oxygen gives us carbon dioxide plus 2 water.



The structure of the DMFC consists of two porous electrolytic electrodes on both sides of a solid polymer electrolyte membrane. The thermodynamic reversible potential for the overall cell reaction is 1.214 volt, Methanol and water are oxidized in the anode catalyst layer (known as ACL) and release electrons and protons according to this reaction. So methanol plus water gives us carbon dioxide + 6 protons + 6 electrons.



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- The electrons are transported through an external circuit to the cathode, while the protons penetrate the electrolyte membrane to the cathode.
- In the *cathode catalyst layer (CCL)*, the oxygen from the ambient atmosphere reacts with electrons and protons and generates water: $6\text{H}^+ + 6\text{e}^- + 1.5\text{O}_2 \rightarrow 3\text{H}_2\text{O}$

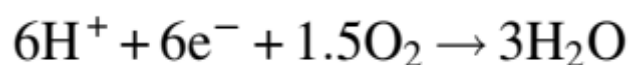
Courtesy: Palombaro et al., Ch. 3, In-Situ Fuel Treatment, Oct 2012.

Courtesy: Indefinivox.com

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You can see how it happens into subsequent representations. This is the overall representation where you can see that this is the chamber. Here you can see that methanol is getting fed to the chamber. And this part is anode, this is cathode. The electron is getting flown like this way from anode to cathode. Then you have to supply oxygen. So, it will result in carbon dioxide this side, water that side and this is a more elaborate way of representation.

Now, these electrons are transported through an external circuit to the cathode, while the protons penetrate the electrolyte membrane to the cathode. So, in the cathode catalyst layer the CCL layer, the oxygen from the ambient atmosphere reacts with electrons and protons and generates water. So 6 protons + 6 electrons + 1.5 moles of oxygen will give us 3 moles of water.



So, you can see here, both are same, here it is written in a better expressive way. So you can see that methanol is getting converted to carbon dioxide. The electron is getting circulated via

the load, from anode to the cathode. This is how it flows, it is very nicely represented there. And the hydrogen is getting transported via this membrane, the solid electrolyte membrane and getting to the other side, where oxygen is getting converted with the help of a proton that is coming from this side to that side and with the help of electrons to water. It is very simplified system and it has a lot of applications.

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- DMFC was initially developed in USA by several organizations including NASA during 1990s.
- It operates at a temperature range of 60 – 130 °C.
- Current DMFCs are limited in the power they can produce, but can still store a high energy content in a small space.
- This means they can produce a small amount of power over a long period of time.
- This makes them ill-suited for powering large vehicles, but ideal for smaller vehicles such as forklifts and tuggers.
- DMFCs are thus used where the power requirement is modest such as mobile electronic devices or chargers and portable power packs.
- Military applications of DMFCs are an emerging application since they have low noise and thermal signatures and no toxic effluent.



Now DMFC was initially developed in the United States by several organizations including NASA during the 1990s. So, it operates at a temperature range of 60 to 130 degrees centigrade. Current DMFCs are limited in the power they can produce (basically they produce small amount of power), but it can still store high energy content in a small space, so that is the beauty of DMFCs. This means that they can produce a small amount of power over a long period of time, sustainability.

This makes them-ill suited for powering large vehicles, but they are very ideal for smaller vehicles such as forklifts and tuggers. Now DMFCs are thus used where the power requirement is modest such as mobile electronic devices or chargers and portable power packs. Military applications of DMFCs are an emerging application since they have low noise and thermal signatures and they have absolutely no toxic effluent.

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(Overview of next lecture)

Module	Module name	Lecture	Title of lecture
11	Organic Commodity Chemicals from Biomass	01	Biomass as feedstock for synthetic organic chemicals, lactic acid, polylactic acid

Thank you

For queries, feel free to contact at: kmohanty@iitg.ac.in



So with this, I wind up today's lecture. So if you have any query, please register it in the Swayam portal or you can always feel free to drop a mail to me at kmohanty@iitg.ac.in. The next module that is module 11 is dedicated to the organic commodity chemicals from biomass. So various types of platform chemicals that are being produced from biomass we will learn about that. And so thank you very much.